Report of November 28, 1973

ANALYSIS OF MAINSTREAM AND SIDESTREAM SMOKE OF CIGARETTES TREATED WITH COLITE

During recent decades, a large number of changes and modifications were introduced and suggested for the reduction of total particulate matter, nicotine, and other toxic compounds in the smoke of cigarettes.[1-4] The suggested and applied modifications include changes in the cigarette paper, especially with respect to porosity and burning rate.[1-7] One patent describes the treatment of cigarette paper with "Colite", a fire retarding material, to reduce particulates in mainstream and sidestream smoke.[7] It was the purpose of this investigation to analyze the mainstream smoke of a popular U.S. cigarette with filter tip for total particulate matter (TPM), nicotine, CO and CO_2 before and after treatment of the cigarette with "Colite". In addition, we also analyzed the sidestream smoke of these cigarettes for TPM and nicotine.

1002802989

EXPERIMENTAL

1. Cigarettes

The National Cancer Institute sent the following cigarettes for testing:

1. 300 popular U.S. cigarettes (85 mm) with filter tip. The filter tip, 30 mm long, consisted of 4 mm overwrap, 11 mm charcoal column (Section I) and 15 mm cellulose acetate column (Section II).

Source: https://www.industrydocuments.ucsf.edu/docs/xfyl0000

2. 00 cigarettes as under (1) but cigarette paper treated with "Colite".

2A. Cigarettes as in (2) but smoking the same number of puffs as Control Cigarettes.

3. 40 popular U.S. cigarettes (85 mm) as under (1); however, 25 mm of the tobacco column was replaced by a cellulose acetate filter tip (total filter length with overwrap 55 mm).

Figure 1 shows cigarettes type 1, 2, and 3.

The cigarettes were stored for 24 hrs in a humidity chamber (Heinr. Borgwaldt; Air Conditioning Chamber) at 22° and at 60% relative humidity. Subsequently, the cigarettes were selected by average weight (± 20 mg of average weight of 200 cigarettes) and stored for another 24 hrs. Cigarettes type 1 and 2 were measured for draw resistance with Filtrona Pressure Drop Tester 3LP[B] at an air flow rate of 17.5 ml/sec. Only those cigarettes were used which were within ± 7% of the draw resistance of 50 cigarettes selected by weight. In the case of cigarette type 3 we had to take the average weight of 40 cigarettes. Since we obtained only 11 cigarettes within ± 20 mg of the average weight, we did not have enough cigarettes to select by draw resistance.

2. Free Burning Rate

Originally we intended to determine, for all cigarettes, the free burning rate of a 40 mm cigarette column using the Filtrona Free Burning Rate Meter, MK-II. During the test, however, we observed that the cigarettes treated with "Colite"

1002802990

Source: https://www.industrydocuments.ucsf.edu/docs/xfyl0000

(types 2 and 3) burnt free for only a limited length. We measured, therefore, only the burning rate of a short tobacco column before it self-extinguished.

3. Smoking of Cigarettes

After 24 hrs of additional storage in the humidity chamber, the cigarettes selected by weight and draw resistance of types 1 and 2 and the cigarettes of type 3 selected by weight only were smoked under standard conditions with a Phipps and Bird 20 channel automatic smoking machine. The conditions were one puff of two seconds once a minute with a puff volume of 35 ml and the following butt lengths:

Cigarette type 1, 33 mm;
Cigarette type 2, 33 mm; and
Cigarette type 2A, butt length was given by the number of puffs it took control cigarette type 1 to reach 33 mm butt length
Cigarette type 3, 58 mm.

4. TPM and Nicotine in Mainstream Smoke

TPM for the mainstream smoke of the cigarettes type 1 and 3 were determined according to the FTC method from four times four cigarettes each.[10] The moisture content of the wet TPM from two times four cigarettes was determined by gas chromatography.[11] In the case of cigarette type 3, we smoked only two times three cigarettes; the moisture content was determined from the TPM of these cigarettes, and the nicotine from the TPM of the other three cigarettes. Nicotine was determined by gas chromatography

1002802991

Source: https://www.industrydocuments.ucsf.edu/docs/xfyl0000

using ^{14}C-labell[illegible] nicotine as internal s[illegible]ndard.[12] In the case of cigarettes type 1, 2, and 2A, nicotine was determined from the TPM of two times four cigarettes.

5. Carbon Monoxide an Carbon Dioxide

Three cigarettes, selected by weight and draw resistance (in case of cigarette type 3, only selected by weight), were smoked under standard conditions through Cambridge filters (CM-113) with a Heinr. Borgwaldt, single channel piston type machine.[14] The gas phase of the individual puffs was blown into a Teflon bag with 1.2 l capacity.[14] From the composite gas phase of all puffs, we injected a small aliquot through a gas dual sampling valve (loop size 0.5 ml) into a 5700A Hewlett-Packard gas chromatograph which was equipped with a thermal conductivity (t.c.) detector. The column eployed measured 6 mm (i.d.) and was packed with the following"sandwich": silica gel (20/100 mesh), 2.0 m; iodine pentoxide, 0.3 m; silver powder, 0.05 m; silica gel, 1.65 m. Within the column, the CO was first separated from CO_2 and, subsequently, oxidized to CO_2. The retention times for CO and CO_2 were 3 and 6 minutes, respectively.

6. TPM and Nicotine in Sidestream Smoke

For the determination of the TPM and nicotine in the sidestream smoke, we smoked the cigarettes under standard conditions in a specially designed smoking chamber (Fig. 2; 15). The air flow through the chamber for sidestream smoke generation was 25 ml per second. We connected the Cambridge filter to the exit port of the chamber for the collection of the sidestream TPM and nicotine.

1002802992

Source: https://www.industrydocuments.ucsf.edu/docs/xfyl0000

From cigarettes type 1, 2, and 2A, we smoked four times four cigarettes and from cigarette type 3 only two cigarettes. The sidestream TPM, moisture content and nicotine were determined as under (5) of the "Experimental" (vide supra).

RESULTS AND DISCUSSION

In Table 1 we have summarized our findings on the mainstream smoke of "Colite" treated cigarettes (type 2), "Colite" treated cigarettes with extended filter tip (type 3), and untreated control cigarettes (type 1). As can be seen from the burning rates as well as from Figure 3 the "Colite" treated cigarettes burn poorly and result in an ash formation which varies greatly from that of commercial cigarettes. The burning rate of the "Colite" treated cigarette was retarded and the draw resistance slightly increased and, therefore, it took about five more puffs to reach the 33 mm butt length compared to the untreated (control) cigarette. As expected, therefore, the "Colite" treated cigarettes deliver more TPM (+ 45%), nicotine (+ 36%), carbon monoxide (+ 56%), and carbon dioxide (+ 50%) than untreated control cigarettes. Comparing 7.9 puffs of the control cigarette (to reach the 33 mm butt length) with 8.0 puffs of the experimental cigarette (2A) we found that the latter was smoked only to a butt length of 51 mm and resulted in reduced values for TPM (- 24%; FTC = TPM, see footnote under Table 1), nicotine (- 19%), carbon monoxide (- 21%), and carbon dioxide (- 22%). We also smoked cigarettes with mannually extended filter tips (type 3). These cigarettes had high draw resistance reflecting high filtration potential

1002802993

Source: https://www.industrydocuments.ucsf.edu/docs/xfyl0000

and were expected to deliver less TPM and nicotine, but relatively high amounts of water, as a result of reverse selectivity.[16] Carbon monoxide and carbon dioxide deliveries were as one may have expected in the smoke of a cigarette with a 27 mm tobacco column.

The sidestream smoke values for TPM and nicotine were significantly lower for the "Colite" treated cigarettes than the untreated cigarettes. This result can be explained, at least in part, by the lower burning rate of the "Colite" treated cigarettes.

In summary, the "Colite" treatment of cigarette paper results in a cigarette which has a low burning rate, requires more puffs to reach standard butt length and, therefore, delivers more "tar" (TPM), nicotine, carbon monoxide and carbon dioxide in the mainstream smoke. Despite the limited data obtained in this study, one may conclude that the "Colite" treatment does not offer an encouraging method for developing the less harmful cigarette.

1002802994

Source: https://www.industrydocuments.ucsf.edu/docs/xfyl0000

REFERENCES

1. Wynder, E.L. and Hoffmann, D.: Tobacco and Tobacco Smoke. Studies in experimental tobacco carcinogenesis. Academic Press, Inc. New York, N.Y., 1967, pp.730

2. Hoffmann, D. and Wynder, E.L.: Selective reduction of tumorigenicity of tobacco smoke. Experimental approaches I and II. Natl. Cancer Inst. Monogr. 28: 151-172, 1968; J. Natl. Cancer Inst., 48: 1849-1868, 1972

3. Müller, R., Moldenhauer, W., and Schlemmer, P.: On the reduction of toxic agents in tobacco smoke (in German) Ber. Inst. Tabakforsch. (Dresden) 15: 126-190, 1968; 16: 116-125, 1969; 17: 66-93, 1970; 18: 95-125, 1971, and 19: 44-59, 1972

4. Wynder, E.L. and Hoffmann, D.: Less harmful ways of smoking. J. Natl. Cancer Inst., 48: 1749-1758, 1972

5. Rickards, J.C. and Owens, W.F., Jr.: Effect of porous paper on yield of the major vapor phase components of cigarette smoke. 20th Tobacco Chemists' Res. Conf., Winston-Salem, N.C., 1966

6. Eitchcock, R.Jr., Cardon, S.Z., and Alvord, E.T.: Cigarette wrapper. U.S. Patent 2,859,753 (1958)

7. Cohn, C.C., U.S. Patent 3,030,963

8. Cigarette Components Ltd., Instrument Division, London, England Filtrona Pressure Drop Tester Mark 4LP, 1972

9. Cigarette Components Ltd., Instrument Division, London, England Filtrona Free-Burning Rate Meter, MKII, 1972

10. Pillsbury, H.C., Bright, C.C., O'Connor, K.J., and Irish, F.W.: Tar and nicotine in cigarette smoke. J. Assoc. Off. Anal. Chem., 111: 458 - 468, 1969

11. Schultz, F.J. and Spears, A.W.: Determination of moisture in total particulate matter. Tobacco Sci., 10: 75076, 1966

12. Rathkamp, G., Tso, T.C., and Hoffmann, D.: Chemical studies on tobacco smoke. XX. Smoke analysis of cigarettes made from Bright tobaccos differing in variety and stalk position. Beitr. Tabakforsch., in print

[Since the method is not yet printed we attach the excerpt for the nicotine method.]

1002802995

Source: https://www.industrydocuments.ucsf.edu/docs/xfyl0000

13. Heinr. Borgwaldt, Hamburg, Germany: Piston-type smoking machine for single cigarettes. 1971

14. Brunnemann, K.D. and Hoffmann, D.: Chemical studies on tobacco smoke. XXIV. A quantitative method for carbon monoxide and carbon dioxide in cigarette and cigar smoke. J. Chrom. Sci., in print.

[Since the method is not yet printed we attach the "Experimental Part" of the paper.]

15. Brunnemann, K.D. and Hoffmann, D.: Chemical studies on tobacco smoke. XXV. On the pH of tobacco smoke. Food Cosmet. Toxic in print.

16. Waltz, P. and Häusemann, M.: Betrachtungen über die Veränderung des Tabakrauches in der Cigarette. Beitr. Tabakforsch., 3: 169-202, 1965

1002802996

Source: https://www.industrydocuments.ucsf.edu/docs/xfyl0000

var ty and stalk positions. . Rathkamp, T.C. Tso, an
. D Hoffmann. Beitr. Tabakf sch., in print

Nicotine

A Cambridge filter packed with the TPM of four cigarettes was placed in a glass vial with 10 ml methanol and shaken mechanically for 2 hrs. Three 10 ml aliquots of the methanol extract were analyzed by gas chromatography on a 4 m column filled with 10% SE-30 on 60/80 mesh Gas Chrom P in conjunction with a flame ionization detector. The injection block temperature was 220° and the column temperature 190°. The average retention times for nicotine and nornicotine were 8.0 and 10.0 min., respectively. The blanks were methanol extracts of unused Cambridge filters. The nicotine values represent an average of two runs of Cambridge filters loaded with the TPM from four cigarettes. The experimental deviation was below ± 5%.

1002802997

Source: https://www.industrydocuments.ucsf.edu/docs/xfyl0000

Excerpt from "Chemical studies on tobacco smoke. XXIV. A quantitative method for carbon monoxide and carbon dioxide in cigarette and cigar smoke." K.D. Brunnemann and D. Hoffmann, J. Chrom. Sci., in print

EXPERIMENTAL

Apparatus: A Borgwaldt piston type smoking machine for single cigarettes used for the analysis is connected with a Hewlett-Packard 5700A gas chromatograph equipped with a dual gas sampling valve No. 18718A, a thermal conductivity detector and a 1 mV recorder (Hewlett-Packard Model 7127A). For the collection of the total gas phase of the mainstream of a cigarette or a cigar, we used a Teflon bag (0.5 and 1.2 l, respectively) with a Swagelok fitting, Teflon ferrules, and Teflon stopcock (custom made by Alltech Associates, Arlington Heights, Ill.). Using the gas chromatographic technique described below, we established the loss by diffusion for CO and CO_2 (Fig. 1) using standard gas mixtures in nitrogen of 1.02% CO and 5.01% CO_2, and 5.07% CO and 10.2% CO_2 (Matheson Gas Products). The loss by diffusion of CO and CO_2 from the gas phase of cigarette smoke were identical with those of the standard gas mixtures. (We did not observe significant differences between individual bags). Before each test series, the bags were reexamined.

Material: To establish the reproducibility of our method, we used a popular U.S. cigarette without filter tip (85 mm). This cigarette and other commercial cigarettes, little cigars, and cigars were bought on the open market in New York City during the second half of 1972. The experimental cigarettes were made available by Dr. T.C. Tso of the U.S. Department of Agriculture, Beltsville, Md., and by Dr. G. Gori of the National Cancer Institute, Bethesda, Md.

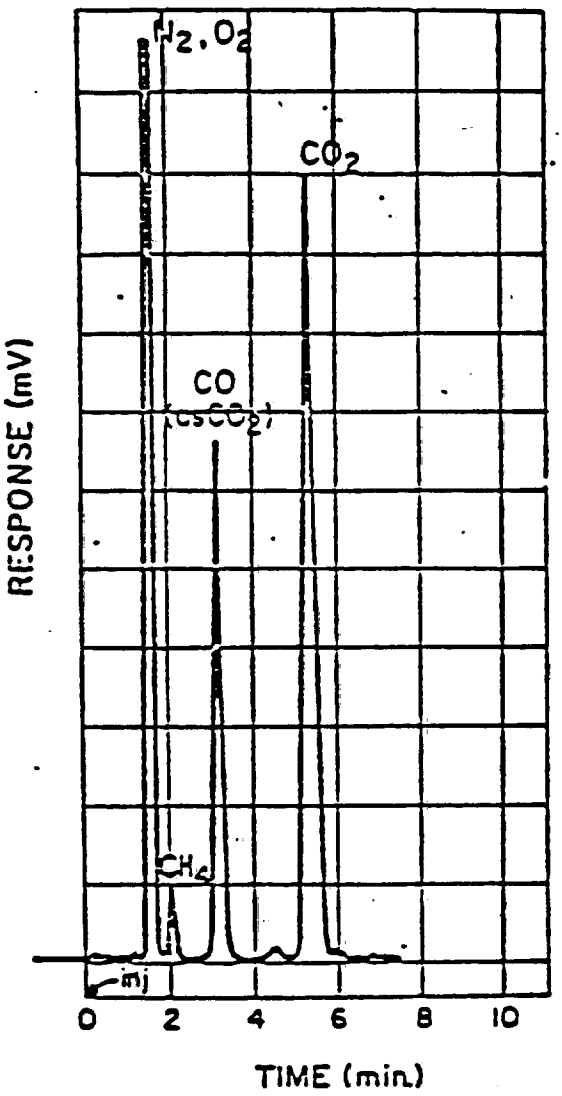
1002802998

Source: https://www.industrydocuments.ucsf.edu/docs/xfyl0000

Gas Chromatography: The separation of CO and CO_2 from other tobacco smoke compounds and the oxidation of CO to CO_2 was accomplished on a mixed packing column. The stainless steel column measured 1/4 inch (o.d.) by 4 m and was packed in such a way that the gas mixture had to pass through 2 m silica gel (28/100 mesh; Fisher), 0.3 m of iodine pentoxide powder (17 g), 0.05 m of silver powder (3.2 g), and finally through 1.65 m of silica gel (28/200 mesh; Fisher; 5, 10, 12). Thus CO was first separated from the CO_2 and, subsequently, oxidized to CO_2 to ensure separation from N_2 (sat. and unsat. hydrocarbons such as methane or isoprene are not oxidized to CO_2 under these conditions). In practical use, the life time of such a column exceeds 1000 injections of tobacco smoke. The gas chromatographic conditions were: carrier gas 50 ml He/min.; oven 130°; thermal conductivity detector 250°, having a bridge current of 225 mA. The retention times for CO (as CO_2) and CO_2 were 3.2 and 5.2 min., respectively (Fig. 2)

1002802999

Source: https://www.industrydocuments.ucsf.edu/docs/xfyl0000



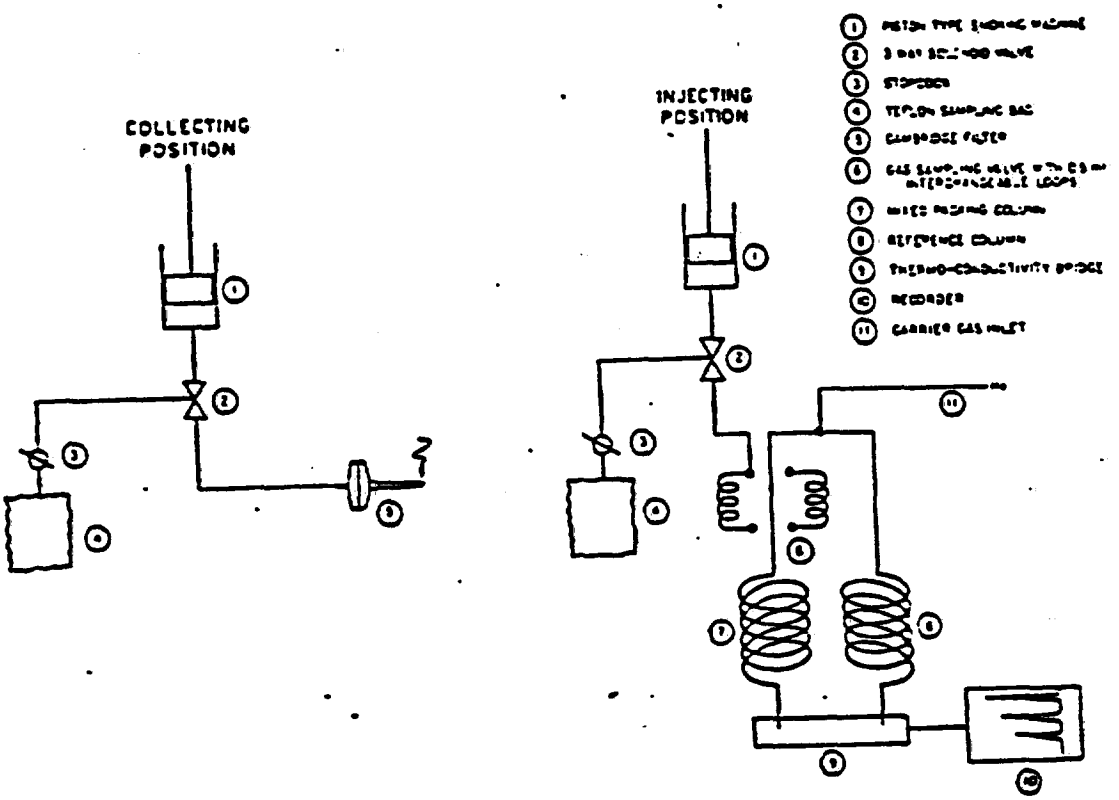
1002803000

Typical chromatograph of CO and CO_2 in cigarette smoke.

EXHIBIT A - AFFIDAVIT OF DR. THOMAS B. OWEN

Source: https://www.industrydocuments.ucsf.edu/docs/xfyl0000

FIGURE 3



Schematic diagram of set-up for the determination of CO and CO_2 in cigarette and cigar smoke

1002803001

EXHIBIT A - AFFIDAVIT OF DR. THOMAS B. OWEN

Source: https://www.industrydocuments.ucsf.edu/docs/xfyl0000

TABLE 1

YIELDS OF MAINSTREAM SMOKE CONSTITUENTS

eight mg)	Total Length (mm)	Butt Length (mm)	Burn. Rate (mg/min.)	Draw Res. (mm)	Puffs per Cig.	Wet TPM (mg/cig.)	Nico-tine (mg/cig.)	Water (mg/cig.)	Dry* TPM (mg)	CO Vol. %	CO_2 Vol. %	CO (mg)	CC (mg
100	85	33	71.0	68	7.9	15.6	1.10	1.91	12.5	3.09	7.69	10.	4
2	85	33	26.1	74	12.9	21.9	1.50	2.16	18.2	3.08	7.37	16.7	6
102	85	51	26.1	74	8.0	12.3	0.89	1.77	9.6	2.65	6.47	8.48	3
116	85	58	19.7	104	6.4	6.7	0.55	1.72	4.4	3.74	8.17	9.46	3

* Federal Trade Commission value for TPM = TPM wet minus water minus nicotine[10]

Type 2A data based on 8 puffs
Type 3, experimental variations ± 20%

EXHIBIT A
AFFIDAVIT OF DR. THOMAS B. OWEN

100280300

Source: https://www.industrydocuments.ucsf.edu/docs/xfyl0000

TABLE 2

TPM AND NICOTINE IN SIDESTREAM SMOKE

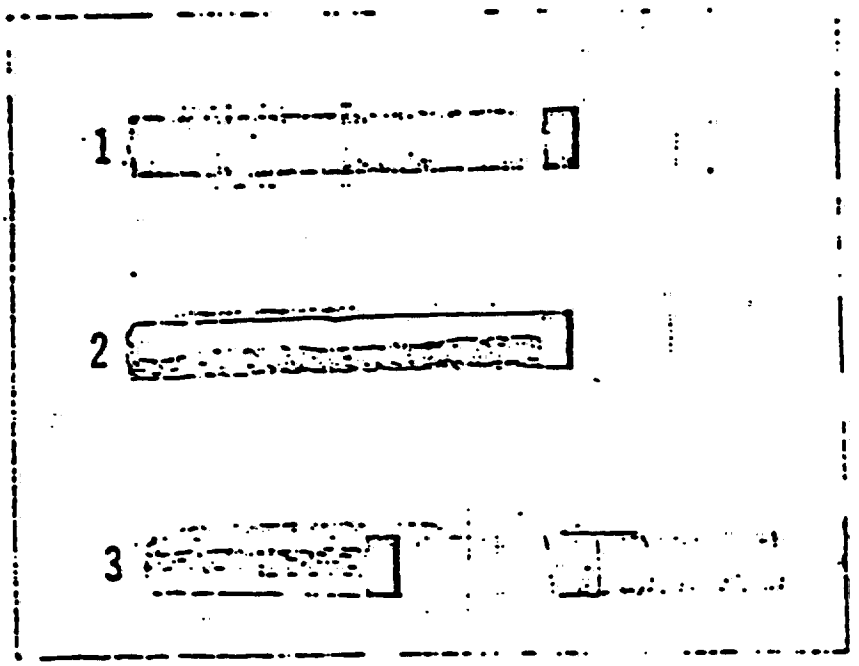
Cigarette Type	Puffs per Cigarette	Wet TPM (mg/cig.)	Nicotine (mg/cig.)	Water (mg/cig.)	Dry TPM * (mg/cig.
1	9.2	20.2	1.59	1.04	17.6
2	13.8	11.5	0.95	0.86	9.7
2A	9.0	8.0	0.90	0.72	7.0
3	7.7	7.4	0.49	0.48	6.4

* See explanation under Table 1

1002803003

Source: https://www.industrydocuments.ucsf.edu/docs/xfyl0000

FIGURE 1

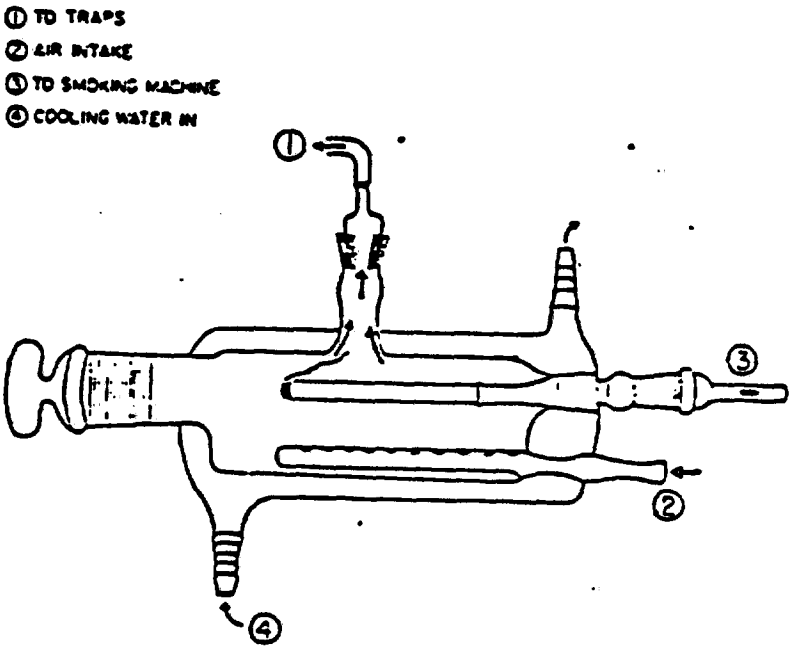


Polaroid photograph of test cigarettes type 1, 2, and 3

1002803004

EXHIBIT A - AFFIDAVIT OF THOMAS B. OWEN

Source: https://www.industrydocuments.ucsf.edu/docs/xfyl0000



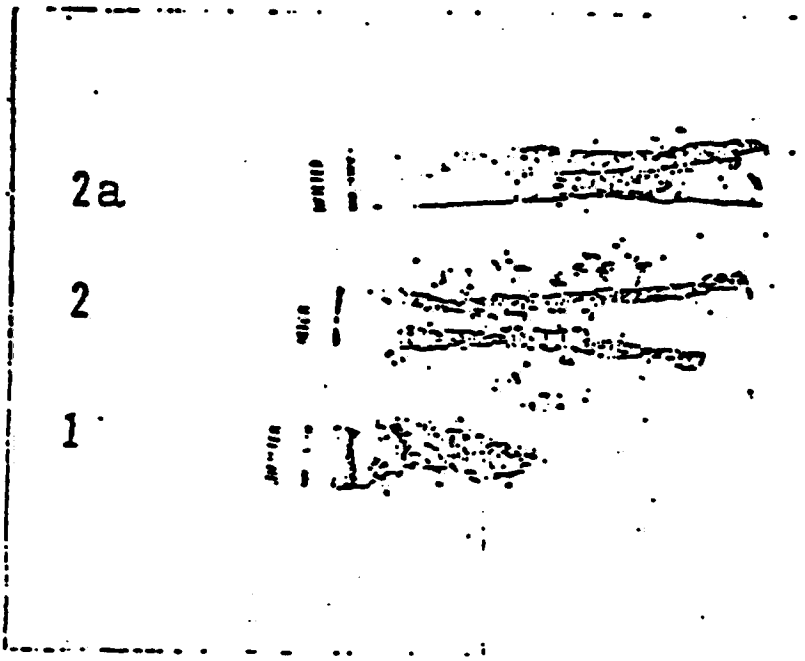
Smoke chamber for determination of TPM and nicotine in sidestream smoke. (Chamber constructed by R.C. Ewald, Inc., Middle Village NY 11379)

1002803005

EXHIBIT A - AFFIDAVIT OF DR. THOMAS B. OWEN

Source: https://www.industrydocuments.ucsf.edu/docs/xfyl0000

FIGURE 3



Partially smoked test cigarettes

1002803006

EXHIBIT A - AFFIDAVIT OF DR. THOMAS B. OWEN

Source: https://www.industrydocuments.ucsf.edu/docs/xfyl0000